

10/586016

***** QUERY RESULTS *****

=> d his l34

(FILE 'WPIX' ENTERED AT 14:55:16 ON 25 NOV 2009)
L34 1 S L30 OR L32 OR L33

=> d que l34

L4 189083 SEA FILE=HCAPLUS ABB=ON PLU=ON HYDROGENATION?
L5 45797 SEA FILE=HCAPLUS ABB=ON PLU=ON "HYDROGENATION CATALYSTS"+UF/C
T
L6 39440 SEA FILE=HCAPLUS ABB=ON PLU=ON UNSAT? (2A) (POLYMER? OR
COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)
L7 23099 SEA FILE=HCAPLUS ABB=ON PLU=ON LINEAR (2A) (POLYMER? OR
COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)
L8 1289 SEA FILE=HCAPLUS ABB=ON PLU=ON (L6 OR L7) (L) (DOUBLE BOND#)

L16 1323 SEA FILE=HCAPLUS ABB=ON PLU=ON METAL COLLOID#
L19 3426 SEA FILE=HCAPLUS ABB=ON PLU=ON COLLOID# AND LATEX?
L24 47 SEA FILE=HCAPLUS ABB=ON PLU=ON L16 AND L19
L30 1 SEA FILE=WPIX ABB=ON PLU=ON L4 AND L19
L32 1 SEA FILE=WPIX ABB=ON PLU=ON L24 AND (L4 OR L5)
L33 1 SEA FILE=WPIX ABB=ON PLU=ON L24 AND L8
L34 1 SEA FILE=WPIX ABB=ON PLU=ON L30 OR L32 OR L33

=> d l34 iall abeq tech abex

L34 ANSWER 1 OF 1 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN
ACCESSION NUMBER: 2005-563958 [57] WPIX
TITLE: Hydrogenation of unsaturated
polymers, e.g. to produce hydrogenated NBR,
comprises making a metal colloid
under reducing conditions in presence of the polymer
latex and then hydrogenating the latex
DERWENT CLASS: A18
INVENTOR: CREUTZ K; WEISS T
PATENT ASSIGNEE: (FARB-C) BAYER AG; (CREU-I) CREUTZ K; (WEIS-I) WEISS T;
(LNXS-C) LANXESS DEUT GMBH
COUNTRY COUNT: 108

PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2005068512	A2	20050728	(200557)*	DE	12[0]	
DE 102004002576	A1	20050804	(200557)	DE		
EP 1709086	A2	20061011	(200667)	DE		
CN 1910208	A	20070207	(200743)	ZH		
US 20080234437	A1	20080925	(200866)	EN		
TW 297695	B1	20080611	(200924)	ZH		

APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2005068512	A2	WO 2005-EP75	20050107
DE 102004002576	A1	DE 2004-102004002576	20040117
CN 1910208	A	CN 2005-80002602	20050107
EP 1709086	A2	EP 2005-700733	20050107
EP 1709086	A2 PCT Application	WO 2005-EP75	20050107

10/586016

US 20080234437 A1 PCT Application WO 2005-EP75 20050107
US 20080234437 A1 US 2006-586016 20060714
TW 297695 B1 TW 2005-101063 20050114

FILING DETAILS:

PATENT NO	KIND	PATENT NO
EP 1709086	A2 Based on	WO 2005068512 A

PRIORITY APPLN. INFO: DE 2004-102004002576 20040117

INT. PATENT CLASSIF.:

MAIN: C08F008-04
SECONDARY: C08F226-08
IPC ORIGINAL: C08C0019-00 [I,C]; C08C0019-00 [I,C]; C08C0019-02 [I,A];
C08C0019-02 [I,A]; C08F0036-00 [I,C]; C08F0036-06 [I,A];
C08F0008-00 [I,C]; C08F0008-04 [I,A]
IPC RECLASSIF.: C08C0019-00 [I,C]; C08C0019-02 [I,A]; C08F0008-00 [I,C];
C08F0008-00 [I,C]; C08F0008-04 [I,A]; C08F0008-04 [I,A]
ECLA: C08F0008-04
USCLASS NCLM: 525/331.900

BASIC ABSTRACT:

WO 2005068512 A2 UPAB: 20090423

NOVELTY - A method for the hydrogenation of unsaturated polymers with double bonds (I) comprises (a) making a metal-containing colloid under reducing conditions in presence of (I) in the form of a latex, (b) hydrogenating the mixture and then separating the colloid from the latex and isolating the polymer.

USE - For the production of hydrogenated diene (co)polymers with improved properties, e.g. partly or completely hydrogenated NBR with better ageing resistance and ozone resistance and lower swelling capacity than the unhydrogenated rubber.

ADVANTAGE - A technically-simple economical method for the hydrogenation of unsaturated polymers to give high-quality products.

MANUAL CODE: CPI: A04-B01D; A10-E13

TECH

POLYMERS - Preferred Polymers: Homopolymers of conjugated dienes or copolymers of 1-5 wt% conjugated diene and 95-99 wt% vinyl group-containing monomers, used in the form of a 1-50 wt% aqueous emulsion (latex).

Preferred Method: Stage (a) is carried out at pH 3-6, using salts or complexes of Group VIIIB metals or ruthenium or rhodium. Stage (b) is carried out at 25-100degreesC and 0.1-100 bar.

ABEX EXAMPLE - A solution of 0.126 g nickel chloride hexahydrate and 0.36 g Mersolat 30 (RTM: alkyl sulfonate) in 10 ml water was treated with 0.5 g N-vinylpyrrolidone/2-dimethylaminoethyl methacrylate copolymer (19 wt% in water), then this mixture was mixed with 20 ml NBR latex (Perbunan NT 3445 (RTM; 19.27 wt%), hydrochloric acid (pH 5) and 5 wt% Mersolat 30 (RTM)(based on solid rubber) and treated slowly with sodium borohydride solution (0.12 g in 5 ml water); after 1 hour all the NaBH4 was added, to give a greyish-black, nickel-containing latex. The fresh latex (40 ml) was then hydrogenated for 3 hours at 10 bar and 65degreesC and worked up, e.g. by oxidation of the nickel with oxygen/citric acid, followed by coagulation of the resulting white latex and isolation of the rubber. With a latex containing 17.7 mmols nickel, this gave a product with a degree of hydrogenation of 12 (0) mol%, a THF-solubles content of 99.2 (37.3) wt%, a solution viscosity (in THF) of 180.6 (82.8) ml/g and a mol. wt. (Mw) of 211100. Values in brackets were obtained if the nickel colloid was prepared at pH 14 instead of pH 5. The original NBR

10/586016

had a solubles content of 99.3 wt%, a viscosity of 146.8 ml/g and a mol. wt. of 128800; a blank test with no nickel gave a polymer with 99.5 wt% solubles and a viscosity of 153 ml/g.

=> d his 129

(FILE 'HCAPLUS' ENTERED AT 14:36:21 ON 25 NOV 2009)

L29 11 S L23 OR L28

=> d que 129

L4 189083 SEA FILE=HCAPLUS ABB=ON PLU=ON HYDROGENATION?
 L5 45797 SEA FILE=HCAPLUS ABB=ON PLU=ON "HYDROGENATION CATALYSTS"+UF/C
 T
 L6 39440 SEA FILE=HCAPLUS ABB=ON PLU=ON UNSAT? (2A) (POLYMER? OR
 COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)
 L7 23099 SEA FILE=HCAPLUS ABB=ON PLU=ON LINEAR (2A) (POLYMER? OR
 COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)
 L8 1289 SEA FILE=HCAPLUS ABB=ON PLU=ON (L6 OR L7) (L) (DOUBLE BOND#)
 L9 30 SEA FILE=HCAPLUS ABB=ON PLU=ON L4 AND L8
 L10 14 SEA FILE=HCAPLUS ABB=ON PLU=ON L5 AND L8
 L11 269 SEA FILE=HCAPLUS ABB=ON PLU=ON (L6 OR L7) (3A) LATEX?
 L12 2 SEA FILE=HCAPLUS ABB=ON PLU=ON (L9 OR L10) AND L11
 L13 4 SEA FILE=HCAPLUS ABB=ON PLU=ON L11 AND COLLOID#
 L14 10503 SEA FILE=HCAPLUS ABB=ON PLU=ON METAL# AND COLLOID#
 L15 1 SEA FILE=HCAPLUS ABB=ON PLU=ON (L9 OR L10) AND L14
 L16 1323 SEA FILE=HCAPLUS ABB=ON PLU=ON METAL COLLOID#
 L17 1 SEA FILE=HCAPLUS ABB=ON PLU=ON L8 AND L16
 L18 1 SEA FILE=HCAPLUS ABB=ON PLU=ON (L9 OR L10) AND COLLOID#
 L19 3426 SEA FILE=HCAPLUS ABB=ON PLU=ON COLLOID# AND LATEX?
 L20 8 SEA FILE=HCAPLUS ABB=ON PLU=ON L4 AND L19
 L21 3 SEA FILE=HCAPLUS ABB=ON PLU=ON L5 AND L19
 L22 14 SEA FILE=HCAPLUS ABB=ON PLU=ON L12 OR L13 OR L15 OR L17 OR
 L18 OR L20 OR L21
 L23 4 SEA FILE=HCAPLUS ABB=ON PLU=ON L22 AND 39/SC, SX
 L28 9 SEA FILE=HCAPLUS ABB=ON PLU=ON L22 AND (METHOD# OR TECHNI?
 OR PROCEDURE? OR PROCESS?)
 L29 11 SEA FILE=HCAPLUS ABB=ON PLU=ON L23 OR L28

=> d his 143

(FILE 'APOLLIT, EMA, RAPRA, SCISEARCH, PASCAL' ENTERED AT 15:01:19 ON 25 NOV 2009)

L43 8 S (L38 OR L41) AND L4
 SAVE TEMP L43 BOY016MULTI/A

FILE 'STNGUIDE' ENTERED AT 15:04:20 ON 25 NOV 2009

FILE 'WPIX' ENTERED AT 15:04:58 ON 25 NOV 2009

FILE 'STNGUIDE' ENTERED AT 15:04:58 ON 25 NOV 2009

=> d que 143

L4 189083 SEA FILE=HCAPLUS ABB=ON PLU=ON HYDROGENATION?
 L6 39440 SEA FILE=HCAPLUS ABB=ON PLU=ON UNSAT? (2A) (POLYMER? OR
 COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)
 L7 23099 SEA FILE=HCAPLUS ABB=ON PLU=ON LINEAR (2A) (POLYMER? OR
 COPOLYMER? OR RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)

10/586016

L8 1289 SEA FILE=HCAPLUS ABB=ON PLU=ON (L6 OR L7) (L) (DOUBLE BOND#)

L16 1323 SEA FILE=HCAPLUS ABB=ON PLU=ON METAL COLLOID#

L19 3426 SEA FILE=HCAPLUS ABB=ON PLU=ON COLLOID# AND LATEX?

L24 47 SEA FILE=HCAPLUS ABB=ON PLU=ON L16 AND L19

L27 1 SEA FILE=HCAPLUS ABB=ON PLU=ON L24 AND L8

L30 1 SEA FILE=WPIX ABB=ON PLU=ON L4 AND L19

L38 8 SEA L30

L41 0 SEA L27

L43 8 SEA (L38 OR L41) AND L4

=> dup rem 129 143

FILE 'HCAPLUS' ENTERED AT 15:05:15 ON 25 NOV 2009
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FILE 'SCISEARCH' ENTERED AT 15:05:15 ON 25 NOV 2009
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 PROCESSING COMPLETED FOR L29
 PROCESSING COMPLETED FOR L43

L44 18 DUP REM L29 L43 (1 DUPLICATE REMOVED)
 ANSWERS '1-11' FROM FILE HCAPLUS
 ANSWERS '12-13' FROM FILE APOLLIT
 ANSWER '14' FROM FILE RAPRA
 ANSWERS '15-16' FROM FILE SCISEARCH
 ANSWERS '17-18' FROM FILE PASCAL

=> d 144 1-11 ibib abs hitind; d 144 12-18 ibib ab ind

L44 ANSWER 1 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2006:935673 HCAPLUS Full-text
 DOCUMENT NUMBER: 147:74062
 TITLE: Ozonization of unsaturated polymers
 in synthetic latexes
 AUTHOR(S): Poluektov, P. T.; Vlasova, L. A.; Shutilin, Yu. F.;
 Korchagin, V. I.
 CORPORATE SOURCE: Voronezh. Fil., FGUP NII Sint. Kauchuka, Voronezh,
 Russia
 SOURCE: Kauchuk i Rezina (2006), (3), 24-27
 CODEN: KCRZAE; ISSN: 0022-9466
 PUBLISHER: Kauchuk i Rezina
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian
 AB Ozonization of NR, butadiene and styrene-butadiene latexes have been studied.
 Amount of ozone reacting with rubber depends primarily on polymer and ozone

concentration Colloid characteristics of the latexes have little effect on the reaction.

CC 39-7 (Synthetic Elastomers and Natural Rubber)

L44 ANSWER 2 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2005:673330 HCAPLUS Full-text

DOCUMENT NUMBER: 143:154720

TITLE: Method for the hydrogenation of unsaturated polymers containing double bonds

INVENTOR(S): Weiss, Thomas; Creutz, Karin

PATENT ASSIGNEE(S): Lanxess Deutschland G.m.b.H., Germany

SOURCE: PCT Int. Appl., 12 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005068512	A2	20050728	WO 2005-EP75	20050107
WO 2005068512	A3	20050909		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
DE 102004002576	A1	20050804	DE 2004-102004002576	20040117
EP 1709086	A2	20061011	EP 2005-700733	20050107
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK, IS			
CN 1910208	A	20070207	CN 2005-80002602	20050107
US 20080234437	A1	20080925	US 2006-586016	20060714
PRIORITY APPLN. INFO.:			DE 2004-102004002576A	20040117
			WO 2005-EP75	W 20050107

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB According to title method, the unsatd. polymer such as nitrile rubber, that is provided in the form of latex, is hydrogenated in the presence of a metal-containing colloid.

IC ICM C08F008-04

CC 39-7 (Synthetic Elastomers and Natural Rubber)

ST metal colloid catalyst hydrogenation nitrile rubber latex

IT Nitrile rubber, preparation

RL: IMF (Industrial manufacture); PREP (Preparation)
(hydrogenated; hydrogenation of unsatd. rubber latexes in presence of metal-containing colloids)
)

IT Colloids

Hydrogenation catalysts
(hydrogenation of unsatd. rubber latexes in presence of metal-containing colloids)

IT Metals, uses

RL: CAT (Catalyst use); USES (Uses)
 (hydrogenation of unsatd. rubber latexes in
 presence of metal-containing colloids)
 IT 7718-54-9, Nickel chloride, uses
 RL: CAT (Catalyst use); USES (Uses)
 (hydrogenation of unsatd. rubber latexes in
 presence of metal-containing colloids)
 IT 9003-18-3DP, hydrogenated
 RL: IMF (Industrial manufacture); PREP (Preparation)
 (nitrile rubber; hydrogenation of unsatd. rubber
 latexes in presence of metal-containing colloids
)
 REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L44 ANSWER 3 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2005:404354 HCAPLUS Full-text

DOCUMENT NUMBER: 144:89234

TITLE: Disperse amphiphilic submicron particles as
 non-covalent supports for cationic homogeneous
 catalysts

AUTHOR(S): Sablong, Rafael; van der Vlugt, Jarl Ivar; Thomann,
 Ralf; Mecking, Stefan; Vogt, Dieter

CORPORATE SOURCE: Schuit Institute of Catalysis, Laboratory of
 Homogeneous Catalysis, Eindhoven University of
 Technology, Eindhoven, 5600, Neth.

SOURCE: Advanced Synthesis & Catalysis (2005), 347(5), 633-636
 CODEN: ASCAF7; ISSN: 1615-4150

PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A simple method for the effective immobilization of homogeneous catalysts on
 polystyrene colloids via non-covalent binding is demonstrated. Stable latexes
 with sufficiently high loading of accessible borate anions are prepared via
 emulsion polymerization Incorporation of cationic rhodium complexes,
 supported via their borate counter-anion is efficient, and these supported
 homogeneous catalysts maintain constant catalytic activity for C:C
 hydrogenation during several recycles, with very low metal leaching.

CC 38-3 (Plastics Fabrication and Uses)

Section cross-reference(s): 67

IT Colloids

(catalyst support; disperse amphiphilic submicron particles as
 non-covalent supports for cationic homogeneous catalysts)

IT Catalyst supports

Hydrogenation

Hydrogenation catalysts

(disperse amphiphilic submicron particles as non-covalent supports for
 cationic homogeneous catalysts)

IT 180420-80-8

RL: CAT (Catalyst use); USES (Uses)

(hydrogenation catalyst; disperse amphiphilic submicron
 particles as non-covalent supports for cationic homogeneous catalysts)

IT 5469-45-4, α -Acetamidocinnamic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(reactant for hydrogenation reaction; disperse amphiphilic
 submicron particles as non-covalent supports for cationic homogeneous
 catalysts)

OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD
 (4 CITINGS)

REFERENCE COUNT: 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS

L44 ANSWER 4 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1998:486370 HCAPLUS Full-text

DOCUMENT NUMBER: 129:175988

ORIGINAL REFERENCE NO.: 129:35773a,35774a

TITLE: Functionalized polymeric and oligomeric
colloids

AUTHOR(S): Rimmer, S.

CORPORATE SOURCE: The Polymer Centre, Lancaster University, Lancaster,
Lanc., LA1 4YA, UKSOURCE: Designed Monomers and Polymers (1998), 1(1), 89-96
CODEN: DMPOF3; ISSN: 1385-772X

PUBLISHER: VSP BV

DOCUMENT TYPE: Journal; General Review

LANGUAGE: English

AB This paper reviews (with 9 refs.) the author's work to date on functional colloids. Colloids with surface grafts of poly(N-isopropylacrylamide) have been prepared by a macromonomer technique. After removal of the anionic surfactant, the colloids displayed temperature-dependent stability. The preparation of functional colloids that are composed of dispersions of telechelic oligomers has been achieved by ozonizing latexes of unsatd. polymers in which the particles were pre-swollen by a hydrophobic solvent. Enzyme immobilization has been carried out by reaction of aldehyde functional colloids with porcine pancreatic lipase. Functional colloids of carboxylic acid-ended oligomer have been further modified with furfuryl amine. The method has been found to be a useful clean technique for the preparation of telechelic oligomers with diene end-groups that can be reacted with dienophile-functionalized polymers. This Diels-Alder reaction yielded amphiphilic networks which may find uses as novel biomaterials. The paper also covers two alternative methods of preparing furan functional colloids of telechelic oligomers: i.e., the use of difuran disulfide as a chain transfer agent in emulsion polymerization and the reaction of functional colloids composed of aldehyde functional telechelic oligomers with furfuryl amine. Finally, some preliminary results on the surface functionalization of polymer colloids via water-tolerant Lewis acids are presented.

CC 35-0 (Chemistry of Synthetic High Polymers)

Section cross-reference(s): 66

ST review functionalized oligomeric polymeric colloid;
polyisopropylacrylamide functionalized colloid review

IT Colloids

(preparation and characterization of functionalized polymeric and oligomeric
colloids)

IT Oligomers

Polymers, preparation

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)

(preparation and characterization of functionalized polymeric and oligomeric
colloids)

IT 25189-55-3P, Poly(N-isopropylacrylamide)

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)

(preparation and characterization of functionalized polymeric and oligomeric
colloids)

OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD
(4 CITINGS)

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L44 ANSWER 5 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1997:683275 HCAPLUS Full-text

DOCUMENT NUMBER: 127:331890

ORIGINAL REFERENCE NO.: 127:65193a,65196a

TITLE: A one-pot method for the preparation of latexes of telechelic oligomers, by ozonolysis of latexes of polymers containing main-chain unsaturation

AUTHOR(S): Ebdon, J. R.; Rimmer, S.

CORPORATE SOURCE: Polymer Centre, School of Physics and Chemistry, Lancaster University, Lancaster, LA1 4YA, UK

SOURCE: Journal of Polymer Science, Part A: Polymer Chemistry (1997), 35(15), 3255-3262
CODEN: JPACEC; ISSN: 0887-624X

PUBLISHER: Wiley

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Poly(Me methacrylate-co-butadiene), poly(Bu methacrylate-co-butadiene) and poly(Me methacrylate-co-2,3-dimethyl butadiene) latexes were prepared by monomer-starved emulsion polymerization. The polymers were followed by GPC. The mol.-weight distribution did not alter significantly with conversion if the polymers were carried out at a feed rate of 0.03 cm³ s⁻¹ per 1000 cm³ of reaction medium and a temperature of 70°. Slower rates of monomer addition led to broadening of the mol.-weight distribution. The resultant latexes were swollen with varying amounts of toluene. Ozonolysis of the swollen and nonswollen latexes yielded latexes of polymer ozonides. Oxidation, with selenium oxide/hydrogen peroxide reagent, converted the ozonides to latexes of carboxylic acid or Me ketone ended telechelic oligomers. The mol. wts. of the oligomers were a function of toluene concentration. Colloidal stability was a function of end-group structure. Thus, carboxylic acid end groups impart extra stability to the colloid while Me ketone end groups do not.

CC 35-8 (Chemistry of Synthetic High Polymers)

Section cross-reference(s): 36, 66

IT Polymerization

(emulsion, monomer-starved; one-pot preparation of telechelic oligomers by ozonolysis of unsatd. polymer latex)

IT Molecular weight distribution

Ozonization

Particle size

(one-pot preparation of telechelic oligomers by ozonolysis of unsatd. polymer latex)

IT Polymers, preparation

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(telechelic, carboxy or ketone-terminated; one-pot preparation of

telechelic

oligomers by ozonolysis of unsatd. polymer latex)

IT 25232-40-0P, 1,3-Butadiene-methyl methacrylate copolymer 32877-24-0P,
2,3-Dimethyl-1,3-butadiene-methyl methacrylate copolymer 43223-25-2P,
1,3-Butadiene-butyl methacrylate copolymer

RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(latex; one-pot preparation of telechelic oligomers by ozonolysis of unsatd. polymer latex)

IT 25232-40-0DP, 1,3-Butadiene-methyl methacrylate copolymer, ozonides, carboxy or ketone terminated 32877-24-0DP, ozonides, carboxy or ketone terminated 43223-25-2DP, 1,3-Butadiene-butyl methacrylate copolymer, ozonides, carboxy or ketone terminated

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(one-pot preparation of telechelic oligomers by ozonolysis of unsatd. polymer latex)

OS.CITING REF COUNT: 15 THERE ARE 15 CAPLUS RECORDS THAT CITE THIS
RECORD (15 CITINGS)
REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L44 ANSWER 6 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1995:294825 HCAPLUS Full-text
DOCUMENT NUMBER: 122:163245
ORIGINAL REFERENCE NO.: 122:30063a,30066a
TITLE: Nitrile group-containing highly saturated copolymer
rubber latex manufacture and latexes thereof
INVENTOR(S): Oora, Kyomori; Mori, Osamu
PATENT ASSIGNEE(S): Nippon Zeon Co, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	----	-----	-----	-----
JP 06287219	A	19941011	JP 1993-97287	19930330
JP 3479995	B2	20031215		

PRIORITY APPLN. INFO.: JP 1993-97287 19930330

AB The title latexes are manufactured by selective hydrogenation of C:C double bonds of nitrile group-containing unsatd. copolymer rubbers in latex state in the presence of added Pd compound solns. containing 0.5-10 mol acid/mol Pd as catalyst. Thus, 400 mL 30%-solid latex containing 37.5:62.5 acrylonitrile-butadiene copolymer rubber prepared by emulsion polymerization in the presence of Na dodecylbenzenesulfonate and 300 mL solution containing Pd acetate and HNO₃ at acid/Pd mol. ratio 1.0 were heated at 50° for 6 h with 30-atm H₂, and condensed to give a latex with I value 64.0 g/100 g, hydrogenation degree 78.2%, and average particle diameter 0.09 µm vs. 270.6, 7.7, and 0.12, resp. for a latex prepared without HNO₃.

IC ICM C08F008-04

ICS C08F220-42

CC 39-7 (Synthetic Elastomers and Natural Rubber)

Section cross-reference(s): 67

ST nitrile rubber hydrogenation; palladium hydrogenation
catalyst nitrile rubber; acidic palladium hydrogenation
catalyst; nitric acid hydrogenation catalyst

IT Hydrogenation catalysts

(acidic palladium solns.; catalysts for hydrogenation of
nitrile group-containing rubber latexes)

IT Rubber, nitrile, preparation

RL: IMF (Industrial manufacture); PREP (Preparation)
(hydrogenated; catalysts for hydrogenation of nitrile
group-containing rubber latexes)

IT Rubber, synthetic

RL: IMF (Industrial manufacture); PREP (Preparation)
(acrylonitrile-butadiene-methacrylic acid, hydrogenated, catalysts for
hydrogenation of nitrile group-containing rubber latexes)

IT 64-19-7, Acetic acid, uses 3375-31-3 3386-65-0, Palladium propionate
7647-10-1, Palladium chloride 7664-38-2, Phosphoric acid, uses
7664-93-9, Sulfuric acid, uses 7697-37-2, Nitric acid, uses
10102-05-3, Palladium nitrate 13566-03-5, Palladium sulfate
13820-53-6, Sodium tetrachloropalladate 19168-23-1, Ammonium
hexachloropalladate
RL: CAT (Catalyst use); USES (Uses)

10/586016

(catalysts for hydrogenation of nitrile group-containing rubber latexes)
IT 9010-81-5P, Acrylonitrile-butadiene-methacrylic acid copolymer
RL: IMF (Industrial manufacture); PREP (Preparation)
(catalysts for hydrogenation of nitrile group-containing rubber latexes)
IT 9003-18-3P
RL: IMF (Industrial manufacture); PREP (Preparation)
(rubber, hydrogenated; catalysts for hydrogenation of nitrile group-containing rubber latexes)
OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD
(1 CITINGS)

L44 ANSWER 7 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1984:456322 HCAPLUS Full-text
DOCUMENT NUMBER: 101:56322
ORIGINAL REFERENCE NO.: 101:8747a,8750a
TITLE: Hydrogenation of carbon-carbon
double bonds in an
unsaturated polymer in latex
form

INVENTOR(S): Wideman, Lawson G.
PATENT ASSIGNEE(S): Goodyear Tire and Rubber Co. , USA
SOURCE: U.S., 6 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 4452950	A	19840605	US 1983-468816	19830222
CA 1216598	A1	19870113	CA 1984-447003	19840208
BR 8400562	A	19840925	BR 1984-562	19840209
EP 117221	A1	19840829	EP 1984-630023	19840217
EP 117221	B1	19891108		
R: BE, DE, FR, GB, IT				
AU 8424775	A	19840830	AU 1984-24775	19840221
AU 559579	B2	19870312		
JP 59161415	A	19840912	JP 1984-32274	19840222
JP 04041161	B	19920707		

PRIORITY APPLN. INFO.: US 1983-468816 A 19830222
ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB A process for the hydrogenation of C-C double bonds of an unsatd. polymer comprises (a) combining the unsatd. polymer in latex form with (1) O, air, or hydroperoxides as oxidizing agents; (2) N₂H₄ or its hydrate as reducing agent; and (3) a metal ion activator and (b) heating the mixture to a temperature from 0° to the reflux temperature of the reaction mixture. Thus, 0.5 mol nitrile rubber (NBR) latex, 0.1 g CuSO₄ initiator, and 100 g N₂H₄.H₂O were charged into a 1-L reactor. Air was charged into the stirred latex mixture at 870 mL/min and the mixture was heated to 60° in the presence of Nalco 2273 defoamer. After 3 h of reaction time, the latex was filtered, coagulated with iso-PrOH, and oven-dried at 50-60°. The H⁺ anal. by NMR showed a 77% reduction of NBR at atmospheric pressure, and IR spectroscopy showed that CN groups were not reduced.

IC C08F008-04
INCL 525339000
CC 39-7 (Synthetic Elastomers and Natural Rubber)
ST hydrogenation double bond unsatd rubber; nitrile rubber

catalytic hydrogenation; hydrazine redn nitrile rubber; copper sulfate catalyst hydrogenation rubber

IT Rubber, butadiene, reactions
 Rubber, butadiene-styrene, reactions
 Rubber, butyl, reactions
 Rubber, isoprene, reactions
 Rubber, natural, reactions
 Rubber, nitrile, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (hydrogenation of latex, catalytic, with hydrazine hydrate)

IT Air
 (in catalytic hydrogenation of unsatd. rubber latexes)

IT Hydrogenation catalysts
 (metal salts, for unsatd. rubber latex)

IT Hydrogenation
 (of carbon-carbon double bonds, in unsatd. rubber latex)

IT Rubber, synthetic
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (butadiene-isoprene, hydrogenation of latex, catalytic, with hydrazine hydrate)

IT 7720-78-7 7758-98-7, uses and miscellaneous
 RL: CAT (Catalyst use); USES (Uses)
 (catalysts, for hydrogenation of unsatd. rubber latexes)

IT 7722-84-1, uses and miscellaneous
 RL: USES (Uses)
 (in catalytic hydrogenation of unsatd. rubber latexes)

IT 7782-44-7, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (in catalytic hydrogenation of unsatd. rubber latexes)

IT 9003-55-8
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (rubber, butadiene-styrene; hydrogenation of latex, catalytic, with hydrazine hydrate)

IT 9003-17-2
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (rubber, butadiene; hydrogenation of latex, catalytic, with hydrazine hydrate)

IT 9010-85-9
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (rubber, butyl; hydrogenation of latex, catalytic, with hydrazine hydrate)

IT 25102-52-7
 RL: USES (Uses)
 (rubber, hydrogenation of, catalytic, with hydrazine hydrate)

IT 9003-31-0
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (rubber, isoprene; hydrogenation of latex, catalytic, with hydrazine hydrate)

IT 9003-18-3
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (rubber, nitrile; hydrogenation of latex, catalytic, with hydrazine hydrate)

OS.CITING REF COUNT: 42 THERE ARE 42 CAPLUS RECORDS THAT CITE THIS RECORD (44 CITINGS)

REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L44 ANSWER 8 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 1931:36936 HCAPLUS Full-text
 DOCUMENT NUMBER: 25:36936

ORIGINAL REFERENCE NO.: 25:4145c-i, 4146a-i, 4147a-i, 4148a-e
 TITLE: Some fundamental rubber problems
 AUTHOR(S): Whitby, G. Stafford
 SOURCE: Transactions, Institution of the Rubber Industry
 (1930), 6, 40-62
 CODEN: TIRIA2; ISSN: 0371-7968
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 GI For diagram(s), see printed CA Issue.
 AB A critical review and discussion, with new points of view and new data, dealing with (1) the chemical constitution of rubber (I) in its relation to the mechanism of polymerization in general, (2) the phys. structure of I in its relation to the behavior of artificial elastic colloids and (3) the nature of vulcanization in its relation to the vulcanization of fatty oils. The weight of evidence on the nature of polymerization favors the idea that in I the polymeric condition is built up through primary valences. Auer (cf. C. A. 23, 4370) has attempted to prove that metastyrene (II) is formed through colloidal forces and distyrene (III) by true polymerization through primary valences, but this is disproved by unpublished expts. of W. and Katz, where it was found that the pyrogenic decomposition of II in vacuo yields III and tristyrene. The mechanism of polymerization suggested by W. and K. (cf. C. A. 22, 1972) is probably typical of unsatd. compds. which form high-mol. polymers, and in general it is characterized by successive addns. to the double bond of the monomer or of the previous polymeric compound with the migration of a H atom to 1 side of the double bond and the residue to the other side. Each stage represents a definite chemical individual. Unpublished expts. show that when polyindenes are decomposed by heat in vacuo, the mol. chain splits at different points (as with II), and mono-, di-, tri- and tetraindene distil, leaving a residual mixture of polyindenes with higher mol. wts. than tetraindene, but lower than the original substance. As with II, the larger the polyindene mol., the greater the ease of cracking. Because of accompanying cyclization, the decomposition of I by heat is not so simple as that of polyindenes; nevertheless it is probable that the mechanism of polymerization of isoprene (IV) to I is similar to that of indene, in which case a formula of I in accord with exptl. evidence can be derived. Any scheme must lead to a very high polymer, which on ozonolysis yields preponderantly levulinic acid (V) and levulinaldehyde (VI). There are 6 possible polymerizations of (x + 2) IV mols., 2 of which ((1) and (2) below) involve 1,2-addition and 4 of which ((3) to (6) below) involve 1,4-addition. At each stage there is a conjugated system like that in the original IV, for all products are IV with a long-chain substituent. (1)

$$\text{H2C:CMech:CH2+HCH:CMech:CH2} \rightarrow \text{H2C:CMechCH2CH2CH:CMech:CH2} \rightarrow +\text{IV}$$

$$\text{H2C:CMechCH2CH2CH:CMechCH2CH2CH:CMech:CH2} \rightarrow +(x-1)\text{IV}$$

$$\text{H2C:CMechCH2CH2[CH:CMechCH2CH2]xCH:CMech:CH2; ozonolysis to (x + 1) mols. V or VI, 2 mols. HCO2H (VII) and 1 mol. AcCO2H (VIII) or AcCHO (IX). (2) H2C:CHCMe:CH2 + HCH:CMech:CH2} \rightarrow \text{H2C:CHCHMeCH2CH:CMech:CH2} \rightarrow +\text{IV}$$

$$\text{H2C:CHCHMeCH2CH:CMechCH2CH2CH:CMech:CH2} \rightarrow +(x-1)\text{IV}$$

$$\text{H2C:CHCHMeCH2[CH:CMechCH2CH2]xCH:CMech:CH2; ozonolysis to x mols. V or VI, 2 mols. VII, 1 mol. VIII or IX and 1 mol. pyrotartaric acid or aldehyde. (3) - CH2CMe:CHCH2- + HCH:CMech:CH2} \rightarrow \text{Me2C:CHCH2CH:CMech:CH2} \rightarrow +x\text{IV}$$

$$\text{Me2C:CHCH2[CH2CMe:CHCH2]xCH:CMech:CH2; ozonolysis to x mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcMe and 1 mol. malonic dialdehyde (X). (4) - CH2CH:CMechCH2 + HCH:CHCMe:CH2} \rightarrow \text{MeCH:CMechCH2CH:CHCMe:CH2} \rightarrow +x\text{IV}$$

$$\text{MeCH:CMechCH2[CH2CH:CMechCH2]xCH:CHCMe:CH2; ozonolysis to x mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcOH or AcH, and 1 mol. AcCH2CO2H or AcCH2CHO. (5) -CH2CMe:CHCH2- + HCH:CHCMe:CH2} \rightarrow \text{Me2C:CHCH2CH:CMechCH2} \rightarrow +\text{IV}$$

$$\text{Me2C:CHCH2CH2CH:CMechCH2CH:CHCMe:CH2} \rightarrow +(x-1)\text{IV}$$

$$\text{Me2C:CHCH2CH2CH:CMechCH2[CH2CH:CMechCH2]xCH:CHCMe:CH2; ozonolysis to (x-1) mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. succinic acid (XI) or aldehyde,$$

and 1 mol. $\text{AcCH}_2\text{CO}_2\text{H}$ or AcCH_2CHO . (6) $-\text{CH}_2\text{CH}:\text{CMeCH}:\text{CMeCH}_2- + \text{HCH}:\text{CMeCH}:\text{CH}_2 \rightarrow \text{MeCH}:\text{CMeCH}_2\text{CH}:\text{CMeCH}:\text{CH}_2 \rightarrow +\text{IV MeCH}:\text{CMeCH}_2\text{CH}_2\text{CMe}:\text{CHCH}_2\text{CH}:\text{CMeCH}:\text{CH}_2 \rightarrow +(\text{x}-1)\text{IV MeCH}:\text{CMeCH}_2\text{CH}_2\text{CMe}:\text{CHCH}_2[\text{CH}_2\text{CMe}:\text{CHCH}_2]\text{xCH}:\text{CMeCH}:\text{CH}_2$; ozonolysis to $(\text{x}-1)$ mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcH or AcOH , 1 mol. acetonylacetone, and 1 mol. X. Any of these is in accord with exptl. data provided x is very large, i. e., where the number of double bonds is too large to distinguish between n and $(\text{n} + 1)$ bonds, for the schemes above require $(\text{n} + 1)$ double bonds per n IV units, and H absorption. Br-addition and mol.-refraction detns. have indicated 1 double bond per IV unit. The ozonolysis products in the schemes above are not incompatible with the numerous expts. of Harries. Of the 6 schemes, present knowledge does not make possible a definite choice. If the addition of successive IV mols. proceeds with the maximum regularity, i. e., involves the same reaction, then a 1,2-addition means scheme (1) and a 1,4-addition scheme (3) or (4). On the contrary, the finding of XI as an ozonolysis product by Harries indicates scheme (5). The expts. of Lebedev on the catalytic hydrogenation of IV suggest that the addition of 1 mol. of IV to another may proceed simultaneously according to schemes (1), (3) and (4), so that rubber is a mixture of isomers, but the asymmetry of IV may restrict the addition to 1 direction. Unpublished expts. by Farmer on the dipolar moment of alkyl-substituted butadienes may throw light on this. The problem might be settled by isolating the initial dimeric and trimeric products. Ostromuiskii (cf. J. Russ. Phys.-Chemical Society 47, 1928(1915)) describes the dimer $\text{H}_2\text{C}:\text{CMeCH}_2\text{CH}_2\text{CH}:\text{CMeCH}:\text{CH}_2$ from IV, but expts. (unpublished) by W. and Crozier, following the directions of O., failed to obtain this dimer. The $-\text{CH}_2\text{CH}:\text{CMeCH}_2[\text{CH}_2\text{CH}:\text{CMeCH}_2]\text{xCH}_2\text{CH}:\text{CMeCH}_2-$ formula of Staudinger and Fritzsche (cf. C. A. 17, 2974) is so out of harmony with the principles of organic chemistry that it cannot be accepted. In spite of the evidence of Stevens (cf. C. A. 13, 3039), it is generally believed that I consists of 2 parts of the same proximate composition. Some of the evidence in favor is not, however, convincing, e. g., the expts. of Bruson, Sebrell and Calvert on the action of SnCl_2 on I (cf. C. A. 21, 3486), where the solvent for separating the product was different from that for separating the original I. A long series of expts. on the swelling of raw I in organic liquids of various types shows that an initial gain in weight resulting from absorption is followed by a continuous loss of weight resulting from diffusion of I from the swollen masses. This diffusion continued, even in relatively poor swelling agents, until all but the protein had dispersed (several yrs. were necessary). This is evidence against the existence of "sol" and "gel" fractions, particularly since the points at which diffusion became very slow in different solvents corresponded to far different proportions of the total I. The proportion which diffuses readily depends upon the swelling power and upon the viscosity of the liquid, and in a given liquid the proportion can be increased by various means, e. g., by the addition of small proportions of strong organic bases and acids, such as piperidine, NH_4Et , NaOEt and NH_3 . Likewise the behavior of vulcanized I in swelling agents gives no evidence of 2 components. Contrary to general belief, vulcanized I diffuses completely in a wide variety of organic liquids (in darkness for several yrs.), e. g., in BzOEt , BuOAc , MeI , CHBr_3 , iso- BuCl , o- $\text{ClC}_6\text{H}_4\text{Me}$, C_6H_6 , EtPrCO , valeric acid, cyclohexanone, hexyl alc., o-tolualdehyde, Pr_2O , di-iso- Bu_2NH , etc. Comparative expts. show that polystyrene (XII) (i. e., autopolymerized styrene and not styrene (XIII) polymerized by heat) and certain other polymers have properties in common with I, e. g., XII resembles I (quant. data are given) in that: (1) both have an "elasticity temperature" below which they are hard; (2) both can be racked; (3) the longer either I or XII is kept stretched, the higher their temperature, and the greater their stretching, the greater are their permanent sets; (4) both behave the same when heated and cooled alternately under a fixed stress; (5) both are stretched to rupture by very small stresses acting over long periods of time, and (6) their sols have the same viscosities under the same conditions. Unlike XII, polystyrene prepared by heating XIII has a measurably low mol. weight, and has a lower elasticity

temperature and smaller elasticity. A product prepared by refluxing XIII for 48 hrs. at 140° had a mol. weight of 1920, and one from heating XIII in a closed vessel for 24 hrs. at 180° had a mol. weight of 2180. Both became soft at 40° and easily stretched at 47°, but with a poor retraction. XII can be made elastic at room temperature by introducing a swelling agent, and recovery after deformation of such swollen XII is more nearly complete than that of XII made elastic by heat, e. g., XII containing 50% Et oleate showed no set after stretching 1300%. XII prepared from solution is extremely elastic and resembles raw rubber, but it gradually loses its elasticity as the solvent evaps., until it becomes friable. A non-volatile solvent (e. g., tricresyl phosphate or iso-Am phthalate) renders XII permanently elastic. Neither XII nor polyvinyl acetate (XIV) is in the form of emulsions or suspensions during its preparation, so the peculiar state of globules in latex is not essential to the peculiar properties of I. Neither is the 2-phase (sol and gel) theory the answer, for XII and XIV are highly heterogeneous, as was shown by fractional precipitation and fractional diffusion. A synthetic methyl rubber, prepared by thermopolymerization of dimethylbutadiene, was separated into fractions of different viscosities in C₆H₆. Natural rubber is probably also very heterogeneous, i. e., it is a mixture of a series of polymers of a wide range of degrees of polymerization; this agrees with its behavior in swelling agents. All fractions disperse ultimately, and though the protein may retard the diffusion, the slowness is due primarily to the high degree of polymerization. This dependence of the rate of diffusion upon the degree of polymerization was also found with vulcanized oils, with diffused I, i. e., I sol (XV) when vulcanized, and with polymethyl acrylate (XVI). The latter, prepared by heating Me acrylate with 0.1% Bz₂O₂ for 0.5 hr. on a water bath, had a mol. weight too high to depress the f. p. of C₆H₆, had a higher degree of swelling than any polymer studied (88.8 times its weight of MeOAc), yet did not disperse in good swelling agents. Direct exptl. evidence of the heterogeneity of raw I was obtained by fractional diffusion, which yielded sols of widely differing viscosities. XV was separated by fractional precipitation into fractions of different properties, and, contrary to Feuchter, Hauser and others, it is very heterogeneous. XV resembles I in that its behavior on mastication is similar, and in that it can be vulcanized (better results are obtained by adding a fatty acid), can be racked, and gives an x-ray diffraction diagram when stretched. Fractional diffusion of I yielded a 1st fraction of 5%, which was an elastic solid, and no evidence was obtained that there is a liquid phase in I. Not only is there a rough parallelism between the average degree of polymerization and the viscosity of a polymer, but for the polymeric products prepared by a given method from a given monomer there probably is a definite degree of polymerization where the elasticity is greatest. Below this, ready deformation is accompanied by poor recovery, and above it the product is tough or brittle. The term "elastic yield value" is suggested as a means of identifying imperfectly elastic colloids, such as I, XII and XIV, and distinguishing them quantitatively from the more nearly perfect elastic colloid, vulcanized rubber. This behavior is complicated at times by an accompanying change from elastic to irreversible deformation, i. e., plastic flow. Furthermore, a permanent set may not result from plastic flow, but from the fact that the elastic yield value is too high at room temperature. The retraction which follows heating rubber under load is probably likewise due to a reduction in the elastic yield value. Swelling agents reduce the elastic yield value of XII, XIV and hydrophilic colloids like silk fibroin and wool keratin. The ultimate explanation of elasticity in organic colloids is not yet in sight. A helical, elastic mol. (cf. Fikentscher and Mark, C. A. 24, 3395) is not an explanation, for widely different mols., e. g., XII, XIV, XVI, sulfurized fatty oils and proteins, are elastic, and inelastic substances are rendered elastic by swelling agents and by heating. Neither does x-ray data so far obtained offer an explanation, and evidence indicates that a pattern means that the substance is becoming inelastic. The concept of an optimal degree of polymerization for maximum elasticity is

fundamental in explaining vulcanization, for the progressive changes when rubber and S are heated probably represent increasing degrees of polymerization. Similarly, when dianisidine and crotonaldehyde (2 mols.) are mixed, the initial product is a thin liquid; on standing at room temperature this becomes viscous, then an elastic solid, then a tough, inelastic solid, and finally a brittle solid; this series of polymerization changes is analogous to those of rubber. The vulcanization of fatty acids throws light on the nature of vulcanization of rubber in that the progressive phys. changes are the same, and both reactions are accelerated by the same organic compds., e. g., dithiocarbamates and mercaptobenzothiazole. Further expts. (cf. W. and Chataway, C. A. 20, 2588) show that accelerators probably act as catalysts of the polymerization. On heating fatty oils, e. g., linseed (XVII), corn, perilla and China wood (XVIII) oils, with S until part had become insol. in acetone but not in Et₂O and on removing all free S, the subsequent addition of rubber vulcanization accelerators decreased the time of heating to cause setting to an elastic solid. Accelerators were much more effective with XVIII than with XVII; this is probably related to the fact that XVIII contains a doubly conjugated linkage system and is converted into a solid by polymerization catalysts like SnCl₄ and SbCl₅, whereas XVII is not thus polymerized. But when heated with S, XVII became polymerizable by SnCl₄ and SbCl₅, probably because of a change in the orientation of the double bonds. By analogy, the vulcanization of I involves essentially a polymerization, and accelerators act as catalysts. As with XVII, a preliminary change by S and heat is necessary. The suggested formula of I (loc. cit.) shows a terminal conjugated system, and it is known that such a system often makes polymerization possible. The vulcanizing action of benzoyl peroxide, a polymerization catalyst, is in accord with the view that vulcanization is essentially polymerization. The aging of vulcanized I probably involves further polymerization by atmospheric O, for the latter is a polymerizing agent. The fact that hydroquinone retards aging is in accord with this view, for it is an anticatalyst of polymerization, e. g., of XIII (private communication).

CC 30 (Rubber and Allied Substances)

IT Colloids

(elastic yield value in identifying imperfectly elastic)

IT Elastic yield value

(in identifying imperfectly elastic colloids)

L44 ANSWER 9 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1931:36935 HCAPLUS Full-text

DOCUMENT NUMBER: 25:36935

ORIGINAL REFERENCE NO.: 25:4145c-i, 4146a-i, 4147a-i, 4148a-e

TITLE: Some fundamental rubber problems

AUTHOR(S): Whitby, G. Stafford

SOURCE: Transactions, Institution of the Rubber Industry
(1929), 5, 184-95

CODEN: TIRIA2; ISSN: 0371-7968

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB A critical review and discussion, with new points of view and new data, dealing with (1) the chemical constitution of rubber (I) in its relation to the mechanism of polymerization in general, (2) the phys. structure of I in its relation to the behavior of artificial elastic ~~colloids~~ and (3) the nature of vulcanization in its relation to the vulcanization of fatty oils. The weight of evidence on the nature of polymerization favors the idea that in I the polymeric condition is built up through primary valences. Auer (cf. C. A. 23, 4370) has attempted to prove that metastyrene (II) is formed through colloidal forces and distyrene (III) by true polymerization through primary valences, but this is disproved by unpublished expts. of W. and Katz, where it

was found that the pyrogenic decomposition of II in vacuo yields III and tristyrene. The mechanism of polymerization suggested by W. and K. (cf. C. A. 22, 1972) is probably typical of unsatd. compds. which form high-mol. polymers, and in general it is characterized by successive addns. to the double bond of the monomer or of the previous polymeric compound with the migration of a H atom to 1 side of the double bond and the residue to the other side. Each stage represents a definite chemical individual. Unpublished expts. show that when polyindenes are decomposed by heat in vacuo, the mol. chain splits at different points (as with II), and mono-, di-, tri- and tetraindene distil, leaving a residual mixture of polyindenes with higher mol. wts. than tetraindene, but lower than the original substance. As with II, the larger the polyindene mol., the greater the ease of cracking. Because of accompanying cyclization, the decomposition of I by heat is not so simple as that of polyindenes; nevertheless it is probable that the mechanism of polymerization of isoprene (IV) to I is similar to that of indene, in which case a formula of I in accord with exptl. evidence can be derived. Any scheme must lead to a very high polymer, which on ozonolysis yields preponderantly levulinic acid (V) and levulinaldehyde (VI). There are 6 possible polymerizations of $(x + 2)$ IV mols., 2 of which ((1) and (2) below) involve 1,2-addition and 4 of which ((3) to (6) below) involve 1,4-addition. At each stage there is a conjugated system like that in the original IV, for all products are IV with a long-chain substituent. (1)

$$\text{H2C:CMech:CH2} + \text{HCH:CMech:CH2} \rightarrow \text{H2C:CMech2CH2CH:CMech:CH2} \rightarrow +\text{IV}$$

$$\text{H2C:CMech2CH2CH:CMech2CH2CH:CMech:CH2} \rightarrow + (x-1)\text{IV}$$

$$\text{H2C:CMech2CH2[CH:CMech2CH2]xCH:CMech:CH2; ozonolysis to } (x + 1) \text{ mols. V or VI, 2 mols. HCO2H (VII) and 1 mol. AcCO2H (VIII) or AcCHO (IX). (2) H2C:CHCMe:CH2} + \text{HCH:CMech:CH2} \rightarrow \text{H2C:CHCHMeCH2CH:CMech:CH2} \rightarrow +\text{IV}$$

$$\text{H2C:CHCHMeCH2CH:CMech2CH2CH:CMech:CH2} \rightarrow + (x-1)\text{IV}$$

$$\text{H2C:CHCHMeCH2[CH:CMech2CH2]xCH:CMech:CH2; ozonolysis to } x \text{ mols. V or VI, 2 mols. VII, 1 mol. VIII or IX and 1 mol. pyrotartaric acid or aldehyde. (3) -}$$

$$\text{CH2CMe:CHCH2-} + \text{HCH:CMech:CH2} \rightarrow \text{Me2C:CHCH2CH:CMech:CH2} \rightarrow +x\text{IV}$$

$$\text{Me2C:CHCH2[CH2CMe:CHCH2]xCH:CMech:CH2; ozonolysis to } x \text{ mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcMe and 1 mol. malonic dialdehyde (X). (4) -}$$

$$\text{CH2CH:CMech2} + \text{HCH:CHCMe:CH2} \rightarrow \text{MeCH:CMech2CH:CHCMe:CH2} \rightarrow +x\text{IV}$$

$$\text{MeCH:CMech2[CH2CH:CMech2]xCH:CHCMe:CH2; ozonolysis to } x \text{ mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcOH or AcH, and 1 mol. AcCH2CO2H or AcCH2CHO. (5) -}$$

$$\text{CH2CMe:CHCH2-} + \text{HCH:CHCMe:CH2} \rightarrow \text{Me2C:CHCH2CH:CMech2} \rightarrow +\text{IV}$$

$$\text{Me2C:CHCH2CH2CH:CMech2CH:CHCMe:CH2} \rightarrow + (x-1)\text{IV}$$

$$\text{Me2C:CHCH2CH2CH:CMech2[CH2CH:CMech2]xCH:CHCMe:CH2; ozonolysis to } (x-1) \text{ mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. succinic acid (XI) or aldehyde, and 1 mol. AcCH2CO2H or AcCH2CHO. (6) -}$$

$$\text{CH2CH:CMech:CMech2-} + \text{HCH:CMech:CH2} \rightarrow \text{MeCH:CMech2CH:CMech:CH2} \rightarrow +\text{IV}$$

$$\text{MeCH:CMech2CH2CMe:CHCH2CH:CMech:CH2} \rightarrow + (x-1)\text{IV}$$

$$\text{MeCH:CMech2CH2CMe:CHCH2[CH2CMe:CHCH2]xCH:CMech:CH2; ozonolysis to } (x-1) \text{ mols. V or VI, 1 mol. VII, 1 mol. VIII or IX, 1 mol. AcH or AcOH, 1 mol. acetylacetone, and 1 mol. X. Any of these is in accord with exptl. data provided } x \text{ is very large, i. e., where the number of double bonds is too large to distinguish between } n \text{ and } (n + 1) \text{ bonds, for the schemes above require } (n + 1) \text{ double bonds per } n \text{ IV units, and H absorption. Br-addition and mol.-refraction detns. have indicated 1 double bond per IV unit. The ozonolysis products in the schemes above are not incompatible with the numerous expts. of Harries. Of the 6 schemes, present knowledge does not make possible a definite choice. If the addition of successive IV mols. proceeds with the maximum regularity, i. e., involves the same reaction, then a 1,2-addition means scheme (1) and a 1,4-addition scheme (3) or (4). On the contrary, the finding of XI as an ozonolysis product by Harries indicates scheme (5). The expts. of Lebedev on the catalytic hydrogenation of IV suggest that the addition of 1 mol. of IV to another may proceed simultaneously according to schemes (1), (3) and (4), so that rubber is a mixture of isomers, but the asymmetry of IV may restrict the addition to 1 direction. Unpublished expts.}$$

by Farmer on the dipolar moment of alkyl-substituted butadienes may throw light on this. The problem might be settled by isolating the initial dimeric and trimeric products. Ostromuiskenski (cf. J. Russ. Phys.-Chemical Society 47, 1928(1915)) describes the dimer $\text{H}_2\text{C}:\text{CMeCH}_2\text{CH}_2\text{CH}:\text{CMeCH}:\text{CH}_2$ from IV, but expts. (unpublished) by W. and Crozier, following the directions of O., failed to obtain this dimer. The $-\text{CH}_2\text{CH}:\text{CMeCH}_2[\text{CH}_2\text{CH}:\text{CMeCH}_2]_x\text{CH}_2\text{CH}:\text{CMeCH}_2-$ formula of Staudinger and Fritsch (cf. C. A. 17, 2974) is so out of harmony with the principles of organic chemistry that it cannot be accepted. In spite of the evidence of Stevens (cf. C. A. 13, 3039), it is generally believed that I consists of 2 parts of the same proximate composition. Some of the evidence in favor is not, however, convincing, e. g., the expts. of Bruson, Sebrell and Calvert on the action of SnCl_2 on I (cf. C. A. 21, 3486), where the solvent for separating the product was different from that for separating the original I. A long series of expts. on the swelling of raw I in organic liquids of various types shows that an initial gain in weight resulting from absorption is followed by a continuous loss of weight resulting from diffusion of I from the swollen masses. This diffusion continued, even in relatively poor swelling agents, until all but the protein had dispersed (several yrs. were necessary). This is evidence against the existence of "sol" and "gel" fractions, particularly since the points at which diffusion became very slow in different solvents corresponded to far different proportions of the total I. The proportion which diffuses readily depends upon the swelling power and upon the viscosity of the liquid, and in a given liquid the proportion can be increased by various means, e. g., by the addition of small proportions of strong organic bases and acids, such as piperidine, NH_4Et , NaOEt and NH_3 . Likewise the behavior of vulcanized I in swelling agents gives no evidence of 2 components. Contrary to general belief, vulcanized I diffuses completely in a wide variety of organic liquids (in darkness for several yrs.), e. g., in BzOEt , BuOAc , MeI , CHBr_3 , iso-BuCl , $\text{o-ClC}_6\text{H}_4\text{Me}$, C_6H_6 , EtPrCO , valeric acid, cyclohexanone, hexyl alc., o-tolualdehyde , Pr_2O , $\text{di-iso-Bu}_2\text{NH}$, etc. Comparative expts. show that polystyrene (XII) (i. e., autopolymerized styrene and not styrene (XIII) polymerized by heat) and certain other polymers have properties in common with I, e. g., XII resembles I (quant. data are given) in that: (1) both have an "elasticity temperature" below which they are hard; (2) both can be racked; (3) the longer either I or XII is kept stretched, the higher their temperature, and the greater their stretching, the greater are their permanent sets; (4) both behave the same when heated and cooled alternately under a fixed stress; (5) both are stretched to rupture by very small stresses acting over long periods of time, and (6) their sols have the same viscosities under the same conditions. Unlike XII, polystyrene prepared by heating XIII has a measurably low mol. weight, and has a lower elasticity temperature and smaller elasticity. A product prepared by refluxing XIII for 48 hrs. at 140° had a mol. weight of 1920, and one from heating XIII in a closed vessel for 24 hrs. at 180° had a mol. weight of 2180. Both became soft at 40° and easily stretched at 47° , but with a poor retraction. XII can be made elastic at room temperature by introducing a swelling agent, and recovery after deformation of such swollen XII is more nearly complete than that of XII made elastic by heat, e. g., XII containing 50% Et oleate showed no set after stretching 1300%. XII prepared from solution is extremely elastic and resembles raw rubber, but it gradually loses its elasticity as the solvent evaps., until it becomes friable. A non-volatile solvent (e. g., tricresyl phosphate or iso-Am phthalate) renders XII permanently elastic. Neither XII nor polyvinyl acetate (XIV) is in the form of emulsions or suspensions during its preparation, so the peculiar state of globules in latex is not essential to the peculiar properties of I. Neither is the 2-phase (sol and gel) theory the answer, for XII and XIV are highly heterogeneous, as was shown by fractional precipitation and fractional diffusion. A synthetic methyl rubber, prepared by thermopolymerization of dimethylbutadiene, was separated into fractions of different viscosities in C_6H_6 . Natural rubber is probably also very heterogeneous, i. e., it is a mixture of a series of polymers of a wide

range of degrees of polymerization; this agrees with its behavior in swelling agents. All fractions disperse ultimately, and though the protein may retard the diffusion, the slowness is due primarily to the high degree of polymerization. This dependence of the rate of diffusion upon the degree of polymerization was also found with vulcanized oils, with diffused I, i. e., I sol (XV) when vulcanized, and with polymethyl acrylate (XVI). The latter, prepared by heating Me acrylate with 0.1% Bz2O2 for 0.5 hr. on a water bath, had a mol. weight too high to depress the f. p. of C6H6, had a higher degree of swelling than any polymer studied (88.8 times its weight of MeOAc), yet did not disperse in good swelling agents. Direct exptl. evidence of the heterogeneity of raw I was obtained by fractional diffusion, which yielded sols of widely differing viscosities. XV was separated by fractional precipitation into fractions of different properties, and, contrary to Feuchter, Hauser and others, it is very heterogeneous. XV resembles I in that its behavior on mastication is similar, and in that it can be vulcanized (better results are obtained by adding a fatty acid), can be racked, and gives an x-ray diffraction diagram when stretched. Fractional diffusion of I yielded a 1st fraction of 5%, which was an elastic solid, and no evidence was obtained that there is a liquid phase in I. Not only is there a rough parallelism between the average degree of polymerization and the viscosity of a polymer, but for the polymeric products prepared by a given ~~method~~ from a given monomer there probably is a definite degree of polymerization where the elasticity is greatest. Below this, ready deformation is accompanied by poor recovery, and above it the product is tough or brittle. The term "elastic yield value" is suggested as a means of identifying imperfectly elastic ~~colloids~~, such as I, XII and XIV, and distinguishing them quantitatively from the more nearly perfect elastic ~~colloid~~, vulcanized rubber. This behavior is complicated at times by an accompanying change from elastic to irreversible deformation, i. e., plastic flow. Furthermore, a permanent set may not result from plastic flow, but from the fact that the elastic yield value is too high at room temperature. The retraction which follows heating rubber under load is probably likewise due to a reduction in the elastic yield value. Swelling agents reduce the elastic yield value of XII, XIV and hydrophilic ~~colloids~~ like silk fibroin and wool keratin. The ultimate explanation of elasticity in organic ~~colloids~~ is not yet in sight. A helical, elastic mol. (cf. Fikentscher and Mark, C. A. 24, 3395) is not an explanation, for widely different mols., e. g., XII, XIV, XVI, sulfurized fatty oils and proteins, are elastic, and inelastic substances are rendered elastic by swelling agents and by heating. Neither does x-ray data so far obtained offer an explanation, and evidence indicates that a pattern means that the substance is becoming inelastic. The concept of an optimal degree of polymerization for maximum elasticity is fundamental in explaining vulcanization, for the progressive changes when rubber and S are heated probably represent increasing degrees of polymerization. Similarly, when dianisidine and crotonaldehyde (2 mols.) are mixed, the initial product is a thin liquid; on standing at room temperature this becomes viscous, then an elastic solid, then a tough, inelastic solid, and finally a brittle solid; this series of polymerization changes is analogous to those of rubber. The vulcanization of fatty acids throws light on the nature of vulcanization of rubber in that the progressive phys. changes are the same, and both reactions are accelerated by the same organic compds., e. g., dithiocarbamates and mercaptobenzothiazole. Further expts. (cf. W. and Chataway, C. A. 20, 2588) show that accelerators probably act as catalysts of the polymerization. On heating fatty oils, e. g., linseed (XVII), corn, perilla and China wood (XVIII) oils, with S until part had become insol. in acetone but not in Et2O and on removing all free S, the subsequent addition of rubber vulcanization accelerators decreased the time of heating to cause setting to an elastic solid. Accelerators were much more effective with XVIII than with XVII; this is probably related to the fact that XVIII contains a doubly conjugated linkage system and is converted into a solid by polymerization catalysts like SnCl4 and SbCl5, whereas XVII is not thus

polymerized. But when heated with S, XVII became polymerizable by SnCl₄ and SbCl₅, probably because of a change in the orientation of the double bonds. By analogy, the vulcanization of I involves essentially a polymerization, and accelerators act as catalysts. As with XVII, a preliminary change by S and heat is necessary. The suggested formula of I (loc. cit.) shows a terminal conjugated system, and it is known that such a system often makes polymerization possible. The vulcanizing action of benzoyl peroxide, a polymerization catalyst, is in accord with the view that vulcanization is essentially polymerization. The aging of vulcanized I probably involves further polymerization by atmospheric O, for the latter is a polymerizing agent. The fact that hydroquinone retards aging is in accord with this view, for it is an anticatalyst of polymerization, e. g., of XIII (private communication).

CC 30 (Rubber and Allied Substances)

IT Colloids

(elastic yield value in identifying imperfectly elastic)

IT Elastic yield value

(in identifying imperfectly elastic colloids)

L44 ANSWER 10 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1928:7395 HCAPLUS Full-text

DOCUMENT NUMBER: 22:7395

ORIGINAL REFERENCE NO.: 22:886e-i,887a

TITLE: Cryoscopic molecular weight determinations of rubber

AUTHOR(S): Pummerer, Rudolf; Nielsen, Hilde; Gundel, Wolfgang

SOURCE: Berichte der Deutschen Chemischen Gesellschaft
[Abteilung] B: Abhandlungen (1927), 60B, 2167-75
CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB Contrary to Staudinger, who considers that macromols. of rubber contain 100-1000 isoprene units, a parent hydrocarbon with a marked tendency to associate leads to mols. of enormous weight. In this case the problem of chemical valence and structure is directly related to ~~colloid~~ chemical and crystallographic problems. An association through secondary valences of the character suggested must be reversible, but it is improbable that the macromol. of Staudinger could dissociate in solution. The results of ultra-violet absorption measurements (cf. preceding abstract) and the relatively high stability of rubber in solution toward I without a catalyst and in the absence of air both preclude the possibility of tervalent C. In view of earlier ~~hydrogenation~~ expts., which indicated a parent mol. with a mol. weight of 600-700, the mol. weight of rubber in camphor was next investigated. Films of rubber were prepared by completely evaporating in vacuo on the walls of a tube at 50° a solution of rubber, and then dissolving directly by admission of fused camphor. This ~~technic~~ gave different results from those obtained by dissolving rubber in camphor, which caused swelling and an apparent solution, the mol. wts. then being very high. At concns. of 1:4 and 1:10, samples of purified rubber, including diffused rubber from ~~latex~~ and crepe, rubber purified by alkali, sol-rubber and gel-rubber showed mol. wts. of 1100-1600. With benzylidenecamphor the mol. wts. were 1400-2500. At concns. of 1:50 in menthol, values of 1200-1600 were obtained. On dilution to 1:100, the mol. wts. were only about 0.5 as great, indicating extensive disassociation. Further dilution did not cause a further decrease in mol. weight. The lowest values for sol-rubber, gel-rubber, and diffused rubber were 520-620, while calcns. based on 8, 9 and 10 isoprene units indicated 544, 612 and 680, resp. The results indicate 8 isoprene units, though the expts. do not warrant the acceptance of this as an established fact. It is of great significance that the rubber fractions which differ so much in solubility show the same average mol. weight in camphor and in menthol. Their essential difference must therefore lie in their different powers of aggregation. The action of O₃, the

light-absorption values and the extraordinary tendency toward cyclization are most readily explained by a ring structure. Hydro-rubber and cyclo-rubber also showed a mol. weight around 600, whereas a partially hydrogenated and a partially cyclized rubber showed values of 1583 and 1442, resp. Under certain conditions a stable "double mol." probably exists, rendering it more difficult still to draw reliable conclusions concerning the mol. weight of rubber.

CC 30 (Rubber and Allied Substances)

L44 ANSWER 11 OF 18 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1927:31483 HCAPLUS Full-text

DOCUMENT NUMBER: 21:31483

ORIGINAL REFERENCE NO.: 21:3765d-i

TITLE: The constitution of rubber

AUTHOR(S): Pummerer, Rudolf

SOURCE: Kautschuk (1927) 233-6

CODEN: KAUTAV; ISSN: 0368-4873

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB A summary, describing in a brief way several fields of investigation, the results of which have not previously been published. To purify rubber, latex was let stand 16 hrs. at 50° with 20% NaOH, the hydrocarbon removed, the process repeated, the NaOH removed by dialysis, and the product fractionated by exhaustive extraction with Et2O. The purified rubber still contains impurities which can be detected by optical means. The gel-rubber is the component responsible for the toughness of rubber and the sol-rubber for the elasticity. When gel-rubber is dissolved in C6H6 and precipitated with EtOH, it becomes partially soluble in Et2O, and becomes more completely soluble if basic or acidic substances like piperidine, EtNH2, NH3 or AcOH are added. Furthermore mastication of gel-rubber in CO2 renders it soluble in Et2O. This suggests that the mol. chain can be ruptured by mech. means though this disaggregation is colloidal or crystallographic, i. e., intramol. rather than intermol. When not stretched, neither gel-rubber nor solrubber show any x-ray evidence of crystallization, but like ordinary rubber they show interference points when stretched. Disaggregation and aggregation are reversible, for among the fractions obtained by evaporating an Et2O solution of rubber are found, after standing, traces of gel-rubber insol. in Et2O. This gel-rubber is rapidly disaggregated by piperidine or NH3. This slow process of aggregation involves the gradual formation of intramol. colloid.-chemical or crystalline arrangements, with accompanying changes in solubility, and is to be distinguished from association. Gel-rubber and sol-rubber probably have the same structural mol., and both these and diffused rubber were found to have mol. wts. of 1100-1600 by the Rast-camphor method. With menthol, in which rubber is readily soluble without swelling, the mol. wts. were 1200-1600 for 2% solns., but only 600 for 0.5-1% solns., the latter value being of significance in connection with a similar value already obtained during hydrogenation expts. and suggested by Hauser and Mark from x-ray studies. Detns. of the osmotic pressure and the diffusion of dilute C6H6 solns. of rubber indicated mol. wts. of 30,000-50,000, and yet sol-rubber can be dialyzed from its C6H6 solution Rubber probably has a parent mol., which by aggregation and disaggregation, association and disassociation, accounts for the various phenomena. The probable organic chemistry of this parent mol. is discussed.

CC 30 (Rubber and Allied Substances)

L44 ANSWER 12 OF 18 APOLLIT COPYRIGHT 2009 FIZ KA on STN DUPLICATE 1

ACCESSION NUMBER: 2005:19300 APOLLIT Full-text

TITLE: Preparation of a polyethylene LaTeX by catalytic hydrogenation of a polybuta-1,4-diene-based dispersion

AUTHOR: Chemtob, Abraham; Heroguez, Valerie; Gnanou, Yves

(Institute for Polymer Materials 'POLYMAT' Grupo de Ingenieria Quimica, Facultad de Ciencias Quimicas, The University of the Basque Country, Apdo 1072, 20080 Donostia-San Sebastian (ES); Laboratoire de Chimie des Polymeres Organiques, UMR5629-CNRS, Universite Bordeaux, Ecole Nationale Superieure de Chimie et de Physique de Bordeaux, 33607 Pessac Cedex (FR))

SOURCE: Macromolecular Rapid Communications (2005) v. 26(21), p. 1711-1715, <http://dx.doi.org/10.1002/marc.200500432>; SICI: 1022-1336(20051101)26:21<1711::AID-MARC200500432>3.0.TX;2-6, With 4 figs., 0 tabs., 20 refs.
CODEN: MRCOE3 ISSN: 1022-1336

DOCUMENT TYPE: Journal
LANGUAGE: English

AB Fully linear polyethylene-based ~~latexes~~ have been prepared by the hydrogenation of polybuta-1,4-diene dispersions. The latter were synthesized via dispersion ring-opening metathesis polymerization of cycloocta-1,5-diene, and hydrogenated using RuCl₂(PPh₃)₃ as catalyst, without any further treatment. A high hydrogenation efficiency was achieved as demonstrated by different techniques including DSC, and ¹H NMR and FT-IR spectroscopy. The hydrogenation process could be carried out without detrimental effect on particle size and colloidal stability as evidenced by optical microscopy and light scattering analysis. (Abstract Copyright [2005], Wiley Periodicals, Inc.)

CC *7220 Dispersions
1511 Polybutadiene
1111 Ethylene polymers
3208 Ring-opening polymerization

CCDE *7220 Dispersionen
1511 Polybutadien
1111 Ethylenpolymere
3208 Polymerisation unter Ringoeffnung

CT CATALYSTS; COLLOIDS; DSC; IR SPECTROSCOPY; LATEX;
LIGHT SCATTERING; MACROMOLECULES, LINEAR; METATHESIS REACTIONS;
MICROSCOPY; NMR; PARTICLE SIZE; POLYBUTADIENE; POLYETHYLENE; RING-OPENING
POLYMERIZATION; RUTHENIUM

CTDE KATALYSTOREN; KOLLOIDE; DSC; IR-SPEKTROSKOPIE; LATEX;
LICHTSTREUUNG; MAKROMOLEKELN, LINEARE; METATHESEREAKTIONEN; MIKROSKOPIE;
NMR; TEILCHENGROESSE; POLYBUTADIEN; POLYETHYLEN; POLYMERISATION UNTER
RINGOEFFNUNG; RUTHENIUM

ST hydrogenation; latex; polybutadiene; polyethylene;
ring-opening metathesis polymerization

L44 ANSWER 13 OF 18 APOLLIT COPYRIGHT 2009 FIZ KA on STN

ACCESSION NUMBER: 1997:8516 APOLLIT Full-text

TITLE: Platinum nanocatalysts immobilized on latex supports

AUTHOR: Mayer, A.B.; Mark, J.E.

SOURCE: J. Polym. Sci., Part B: Polym. Phys. (1997) 35(8), p.1207-1216, 10p, 4f, 7t, 24l
CODEN: JPBPEM ISSN: 0887-6266

DOCUMENT TYPE: Journal
LANGUAGE: English

AB Several ~~latex~~ dispersions of different hydrophobicity were investigated with respect to their ability to adsorb platinum nanoparticles that had been reduced in their presence. The latices used were poly(N-vinyl-2-pyrrolidone-styrene), poly(vinyl chloride-methyl acrylate), PVC, PVAc, PS, poly(N-vinyl-2-pyrrolidone), poly(diallyldimethyl ammonium chloride), and poly(styrene sulfonic acid). Two reduction methods were tested, specifically the slower

method of refluxing the alcoholic solutions and the more rapid method of reaction with KBH₄. The immobilization of the metal particles and their nanosize dimensions were demonstrated by TEM, and their catalytic activity was tested by the hydrogenation of cyclohexene as a model reaction. Some additional immobilized platinum nanoparticles were prepared in the presence of various protective polymers. This can lead to various advantages with respect to, for instance, the stability and the catalytic properties of these materials. Even in the presence of such additional protective polymers, the platinum nanoparticles remained immobilized for some of the hydrophobic latexes both before and after catalytic hydrogenations. (author abstract).

CC *8138 Polymeric agents
1100 Thermoplastics
3230 Catalysts
5120 Dispersions

CCDE *8138 Polymere Agenzien
1100 Thermoplaste
3230 Katalysatoren
5120 Dispersionen

CT ADDITION; ADSORPTION; ALLYL; ALLYL POLYMERS; AMMONIUM COMPOUNDS; CATALYSTS; COLLOIDS; COPOLYMERS; DISPERSIONS; ELECTRON MICROSCOPY; ELEMENTS; HYDROGEN; HYDROGEN CHLORIDE; HYDROPHOBIC; IMMOBILIZATION; METHYL; METHYL ACRYLATE; MORPHOLOGY; PLATINUM; POLYMERIC CATALYSTS; POLYSALTS; POLYSTYRENE; POLYSTYRENE, SULFONATED; POLYVINYL ACETATE; POLYVINYL CHLORIDE; POLYVINYLPIRROLIDONE; REACTIONS WITH LOW MOLECULAR COMPOUNDS; REACTIVITY; REDUCTION; STYRENE; VINYL CHLORIDE; VINYLPIRROLIDONE

CTDE ADDITION; ADSORPTION; ALLYL; ALLYLPOLYMERE; AMMONIUMVERBINDUNGEN; CHLORWASSERSTOFF; COPOLYMERE; DISPERSIONEN; ELEKTRONENMIKROSKOPIE; ELEMENTE; HYDROPHOB; IMMOBILISIERUNG; KATALYSATOREN; KOLLOIDE; METHYL; METHYLACRYLAT; MORPHOLOGIE; PLATIN; POLYMERER KATALYSATOR; POLYSALZE; POLYSTYROL; POLYSTYROL, SULFONIERTES; POLYVINYLACETAT; POLYVINYLCHLORID; POLYVINYLPIRROLIDON; REAKTIONEN AN NIEDERMOLEKULAREN; REAKTIVITAET; REDUKTION; STYROL; VINYLCHLORID; VINYLPIRROLIDON; WASSERSTOFF

ST nanocatalyst; Pt colloid; polymeric carrier; NANOSTRUCTURES

L44 ANSWER 14 OF 18 RAPRA COPYRIGHT 2009 RAPRA on STN

ACCESSION NUMBER: R:599109 RAPRA Full-text
FILE SEGMENT: Rapra Abstracts
TITLE: INTERACTION BEHAVIOUR IN A BINARY MIXTURE OF POLYMER PARTICLES.
AUTHOR: Ottewill R H; Rennie A R (Bristol,University; Cambridge,University)
SOURCE: Progress in Colloid & Polymer Science Vol.100, 1996, p.60-3
ISSN: 0340-255X
CODEN: PCPSD7
PUBLICATION YEAR: 1996
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The behaviour of small charge-stabilised particles in the presence of larger particles in a sodium chloride solution was investigated using small-angle neutron scattering. Data are given for hydrogenated and deuterated PS latex particles. 14 refs.

AN R:599109 RAPRA FS Rapra Abstracts Full-text

CC 42C21; 6121; 611; 94T

SC *QA; UF; OB; KF

CT CALCULATION; CHEMICAL MODIFICATION; COLLOID; DATA; DEUTERATION; EMULSION POLYMERISATION; GRAPH; HYDROGENATION; LATEX; NEUTRON DIFFRACTION; NEUTRON SCATTERING; PARTICLE SIZE; PLASTIC; POLYMERISATION MECHANISM; POLYSTYRENE; PS; SOLUTION; TABLES; TECHNICAL;

TEST; THEORY; THERMOPLASTIC; EMULSION POLYMERIZATION; POLYMERIZATION
MECHANISM

NPT SODIUM CHLORIDE

SHR COLLOIDAL PROPERTIES,PS ~~latex~~; CHEMICAL MODIFICATION,PS
~~latex~~; LATEX,PS,chemical modification,colloidal
properties

GT EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

L44 ANSWER 15 OF 18 SCISEARCH COPYRIGHT (c) 2009 The Thomson Corporation on
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ACCESSION NUMBER: 2003:882570 SCISEARCH Full-text

THE GENUINE ARTICLE: 717XV

TITLE: Synthesis and characterization of palladium containing
membranes based upon polyacrylic acid

AUTHOR: Schomacker R (Reprint)

CORPORATE SOURCE: Tech Univ Berlin, Inst Tech Chem, Str 17 Juni 124, Sekr TC
8, D-10623 Berlin, Germany (Reprint)

AUTHOR: Beyer A; Reichert K H

CORPORATE SOURCE: Tech Univ Berlin, Inst Tech Chem, D-10623 Berlin, Germany

COUNTRY OF AUTHOR: Germany

SOURCE: COLLOID AND POLYMER SCIENCE, (SEP 2003) Vol. 281, No. 9,
pp. 862-868.

ISSN: 0303-402X.

PUBLISHER: SPRINGER-VERLAG, 175 FIFTH AVE, NEW YORK, NY 10010 USA.

DOCUMENT TYPE: Article; Journal

LANGUAGE: English

REFERENCE COUNT: 26

ENTRY DATE: Entered STN: 24 Oct 2003

Last Updated on STN: 24 Oct 2003

ABSTRACT IS AVAILABLE IN THE ALL AND IALL FORMATS

AB Different methods are described to synthesize a highly porous polymer
membrane with fine dispersed metal-nanoparticles. The preparation of the
porous catalytic membranes happens by crosslinking of polyacrylic acid
dispersions with bifunctional crosslinker in presence of palladium
particles. Palladium-nanoparticles, stabilized with the block copolymer
polystyrene-block-polyethyleneoxide, can be immobilized in the polymer
network in different ways. The polymer/metal network can be prepared in
the form of thin flat membranes and dried under retention of the porosity
and three-dimensional network structure. Different reduction and
preparation methods were applied in order to obtain differences in particle
size and distribution of the palladium. The morphology of the material was
investigated by scanning electron microscopy. Transmission electron
microscopy was employed to show the size and distribution of the metal-
nanoparticles in the polymeric matrix. The catalytic activity of the
obtained membranes was investigated for the gas phase hydrogenation of
cyclohexene and propyne.

CC CHEMISTRY, PHYSICAL; POLYMER SCIENCE

ST Author Keywords: polymeric dispersions; interparticle crosslinking;
palladium nanoparticles; catalytic membranes

STP KeyWords Plus (R): BLOCK-COPOLYMER MICELLES; POROUS CERAMIC MEMBRANES;
NOBLE-METAL COLLOIDS; CATALYTIC REACTORS; PD NANOPARTICLES;
LAYERED SILICATE; LATEX SUPPORTS; POLYMERIZATION; CLUSTERS; GEL

ABSTRACT IS AVAILABLE IN THE ALL AND IALL FORMATS

L44 ANSWER 16 OF 18 SCISEARCH COPYRIGHT (c) 2009 The Thomson Corporation on
STN

ACCESSION NUMBER: 1999:608660 SCISEARCH Full-text

THE GENUINE ARTICLE: 226DW

TITLE: Immobilization of palladium nanoparticles on ~~latex~~
supports and their potential for catalytic applications

AUTHOR: Mayer A B R (Reprint)
 CORPORATE SOURCE: Polytech Univ, Dept Chem Engr Chem & Mat Sci, 6 Metrotech Ctr, Brooklyn, NY 11201 USA (Reprint)
 AUTHOR: Mark J E
 CORPORATE SOURCE: Univ Cincinnati, Dept Chem, Cincinnati, OH 45221 USA; Univ Cincinnati, Ctr Polymer Res, Cincinnati, OH 45221 USA
 COUNTRY OF AUTHOR: USA
 SOURCE: ANGEWANDTE MAKROMOLEKULARE CHEMIE, (JUL 1999) Vol. 268, pp. 52-58.
 ISSN: 0003-3146.
 PUBLISHER: WILEY-V C H VERLAG GMBH, PO BOX 10 11 61, D-69451 BERLIN, GERMANY.
 DOCUMENT TYPE: Article; Journal
 LANGUAGE: English
 REFERENCE COUNT: 29
 ENTRY DATE: Entered STN: 1999
 Last Updated on STN: 1999

ABSTRACT IS AVAILABLE IN THE ALL AND IALL FORMATS

AB Palladium nanoparticles were reduced in the presence of several latex dispersions possessing different hydrophobicities. Various reduction methods were investigated, specifically the slower methods of refluxing the alcoholic solution and the more rapid reduction by potassium tetrahydridoborate. In several cases the latexes showed the ability to adsorb and immobilize the palladium nanoparticles on their surface. Transmission electron microscopy was employed to show the immobilization of the metal nanoparticles on the latex surfaces, and their nanosize dimensions. The latex-metal dispersions showed catalytic activity for the hydrogenation of cyclohexene as a model reaction. A selection of water-soluble protective polymers was included to explore whether the metal nanoparticles were still immobilized. In the case of the more hydrophobic latexes, the accumulation and immobilization of the metal nanoparticles was preserved both before and after their use as hydrogenation catalysts.

CC POLYMER SCIENCE

STP KeyWords Plus (R): AMPHIPHILIC BLOCK-COPOLYMERS; METAL POLYMER COMPOSITES; PLATINUM NANOCATALYSTS; HYDROGENATION; COLLOIDS; MICELLES; NANOCLOUDS; PARTICLES; FILMS; SIZE

ABSTRACT IS AVAILABLE IN THE ALL AND IALL FORMATS

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 on STN

ACCESSION NUMBER: 2006-0398467 PASCAL Full-text

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TITLE (IN ENGLISH): Stabilization of rhodium colloids with catalytic properties with ligands or polymers

TITLE (IN FRENCH): Stabilisation de colloides de rhodium a proprietes catalytiques par des ligands ou des polymeres
 Dispersabilite : particules, charges, pigments, latex

AUTHOR: BORSLA Abdou; CANSELIER Jean Paul; WILHELM Anne-Marie; DELMAS Henri

FOISSY Alain (ed.); BACHIRI S.A. (ed.)

CORPORATE SOURCE: Laboratoire de Genie Chimique - UMR CNRS 5503 (INPT/UPS) - Ecole Nationale Supérieure des Ingenieurs en Arts Chimiques et Technologiques - 18 Chemin de la Loge, 31078 Toulouse, France

SOURCE: Cahiers de Formulation, (2003), 251-265, 45 refs.
 Conference: 8 Societe Francaise de Chimie. Groupe Formulation. Journees annuelles, Besancon (France), 17 Oct 2000

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 AVAILABILITY: French
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 CC 001C01A03; Chemistry; General chemistry, Physical chemistry; Catalysis
 001C01J02; Chemistry; General chemistry, Physical chemistry; Colloidal
 state, Dispersed states
 CCFR 001C01A03; Chimie; Chimie generale, Chimie physique; Catalyse
 001C01J02; Chimie; Chimie generale, Chimie physique; Etat colloidal,
 Etats disperses
 CCES 001C01A03; Quimica; Quimica general, Fisicoquimica; Catalisis
 001C01J02; Quimica; Quimica general, Fisicoquimica; Estado coloidal,
 Estados dispersados
 CT Stabilization; Colloid; Rhodium; Platinoid; Catalyst activity;
 Polymer; Organic ligand; Catalytic reaction; ~~Hydrogenation~~;
 Alkene; Hydrocarbon; Heterogeneous catalysis; Nanoparticle;
 Pyrrolidone(vinyl) polymer; Polyvinylalcohol; Phosphine
 CTFR Stabilisation; Colloide; Rhodium; Platinoide; Activite
 catalytique; Polymere; Coordinat organique; Reaction catalytique;
~~Hydrogenation~~; Alcene; Hydrocarbure; Catalyse heterogene;
 Nanoparticule; Pyrrolidone(vinyl) polymere; Vinylique alcool polymere;
 Phosphine; Oct-1-ene
 CTES Estabilizacion; Coloide; Rodio; Platinoide; Actividad catalitica;
 Polimero; Ligando organico; Reaccion catalitica; Hidrogenacion; Alqueno;
 Hidrocarburo; Catalisis heterogenea; Nanoparticula; Pirrolidona(vinil)
 polimero; Vinilico alcohol polimero; Fosfina
 BT Transition metal
 BTFR Metal transition
 BTES Metal transicion
 L44 ANSWER 18 OF 18 PASCAL COPYRIGHT 2009 INIST-CNRS. ALL RIGHTS RESERVED.
 on STN
 ACCESSION NUMBER: 2001-0317756 PASCAL Full-text
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 reserved.
 TITLE (IN ENGLISH): Metal nanocatalysts stabilized in protective polymer
 matrices
 Proceedings of the 5th International Conference on
 Frontiers of Polymers and Advanced Materials, Poznan,
 Poland, June 21-25, 1999. Part II of II
 AUTHOR: MAYER Andrea B. R.; MARK James E.
 PRASAD Paras N. (ed.); KAJZAR F. (ed.); KOZLOWSKI
 Ryszard (ed.)
 CORPORATE SOURCE: Department of Chemistry, University of Cincinnati,
 Cincinnati, OH 45221-0172, United States
 State University of New York at Buffalo, United
 States; LETI-CEA (Technologies Avancees), Saclay,
 France; Institute of Natural Fibres, Poznan, Poland
 SOURCE: Molecular crystals and liquid crystals science and
 technology. Section A, Molecular crystals and liquid
 crystals, (2000), 354, 221-237, 18 refs.
 Conference: 5 International Conference on Frontiers of
 Polymers and Advanced Materials, Poznan (Poland), 21
 Jun 1999
 ISSN: 1058-725X
 DOCUMENT TYPE: Journal; Conference

BIBLIOGRAPHIC LEVEL: Analytic
 COUNTRY: United States
 LANGUAGE: English
 AVAILABILITY: INIST-12857, 354000098187260210

AB Palladium and platinum nanoparticles were generated in the presence of a variety of protective polymers. The size and size distribution of the metal particles were determined by transmission electron microscopy, and the catalytic activities of the metal-polymer systems were qualitatively tested by the ~~hydrogenation~~ of cyclohexene as a model reaction. Several groups of protective polymers were examined, including water-soluble homopolymers and random copolymers, cationic polyelectrolytes, amphiphilic block copolymers, and latices as carriers. The catalytic properties were found to be significantly influenced by the type of protective polymer used.

AN 2001-0317756 PASCAL Full-text

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CC 001D09D04I; Applied sciences; Physicochemistry of polymers, Macromolecular chemistry, Materials science; Organic polymers

CCFR 001D09D04I; Sciences appliquees; Physicochimie des polymeres, Chimie macromoleculaire, Science des materiaux; Polymeres organiques

CCES 001D09D04I; Ciencias aplicadas; Fisicoquimica de los polimeros, Quimica macromolecular, Ciencia de los materiales; Polimeros organicos

CT Polymer; Polyelectrolyte; Amphiphilic polymer; ~~Latex~~; Catalyst support; Metal particle; ~~Colloid~~ particle; Coated particle; Particle size; Catalyst activity; Chemical reduction; Alkene; Comparative study; Experimental study

CTFR Polymere; Polyelectrolyte; Polymere amphiphile; ~~Latex~~; Support catalyseur; Particule metallique; Particule colloïdale; Particule enrobee; Dimension particule; Activite catalytique; Reduction chimique; Alcene; Etude comparative; Etude experimentale; Nanoparticule

CTES Polimero; Polielectrolito; Polimero amfifilo; ~~Latex~~; Soporte catalizador; Particula metalica; Particula coloidal; Particula envuelta; Dimension particula; Actividad catalitica; Reduccion quimica; Alqueno; Estudio comparativo; Estudio experimental

10/586016

***** SEARCH HISTORY *****

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(FILE 'HOME' ENTERED AT 14:32:44 ON 25 NOV 2009)

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L3 1 SEA ABB=ON PLU=ON 9003-18-3/RN
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L5 45797 SEA ABB=ON PLU=ON "HYDROGENATION CATALYSTS"+UF/CT

L6 39440 SEA ABB=ON PLU=ON UNSAT? (2A) (POLYMER? OR COPOLYMER? OR
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L7 23099 SEA ABB=ON PLU=ON LINEAR (2A) (POLYMER? OR COPOLYMER? OR
RESIN# OR TERPOLYMER# OR HOMOPOLYMER?)

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L10 14 SEA ABB=ON PLU=ON L5 AND L8

L11 269 SEA ABB=ON PLU=ON (L6 OR L7) (3A) LATEX?

L12 2 SEA ABB=ON PLU=ON (L9 OR L10) AND L11
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L13 4 SEA ABB=ON PLU=ON L11 AND COLLOID#
D SCA TI HIT

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L19 3426 SEA ABB=ON PLU=ON COLLOID# AND LATEX?

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L21 3 SEA ABB=ON PLU=ON L5 AND L19
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D SCA L21

L22 14 SEA ABB=ON PLU=ON L12 OR L13 OR L15 OR L17 OR L18 OR L20 OR
L21

L23 4 SEA ABB=ON PLU=ON L22 AND 39/SC, SX
D SCA TI HIT

L24 47 SEA ABB=ON PLU=ON L16 AND L19

L25 2 SEA ABB=ON PLU=ON L24 AND (L4 OR L5)

L26 5 SEA ABB=ON PLU=ON L23 OR L25

L27 1 SEA ABB=ON PLU=ON L24 AND L8
D SCA TI

L28 9 SEA ABB=ON PLU=ON L22 AND (METHOD# OR TECHNI? OR PROCEDURE?
OR PROCESS?)

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10/586016

L32 1 SEA ABB=ON PLU=ON L24 AND (L4 OR L5)
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L34 1 SEA ABB=ON PLU=ON L30 OR L32 OR L33
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L35 8 SEA ABB=ON PLU=ON L11 AND COLLOID#
D SCA
L36 0 SEA ABB=ON PLU=ON L35 AND L4
L37 0 SEA ABB=ON PLU=ON L35 AND HYDROGEN?
D TI KWIC L34
D L34 AU

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L39 0 SEA ABB=ON PLU=ON L21
L40 1 SEA ABB=ON PLU=ON L25
L41 0 SEA ABB=ON PLU=ON L27
L42 0 SEA ABB=ON PLU=ON L13
L43 8 SEA ABB=ON PLU=ON (L38 OR L41) AND L4
D TI KWIC
D TI KWIC 2-3
SAVE TEMP L43 BOY016MULTI/A

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D QUE L34

FILE 'WPIX' ENTERED AT 15:04:58 ON 25 NOV 2009
D L34 IALL ABEQ TECH ABEX

FILE 'STNGUIDE' ENTERED AT 15:04:58 ON 25 NOV 2009
D QUE L29
D QUE L43

FILE 'HCAPLUS, APOLLIT, RAPRA, SCISEARCH, PASCAL' ENTERED AT 15:05:15 ON
25 NOV 2009

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ANSWERS '12-13' FROM FILE APOLLIT
ANSWER '14' FROM FILE RAPRA
ANSWERS '15-16' FROM FILE SCISEARCH
ANSWERS '17-18' FROM FILE PASCAL
D L44 1-11 IBIB ABS HITIND
D L44 12-18 IBIB AB IND